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DOCKET NO. ORT-1230

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:

S. Dax et al.

Serial No.:

09/552,969

Art Unit:

1624

Filed

April 20, 2000

Examiner:

Hong Liu

Title

3a,4,5,9b-tetrahydro-1h-benz[e]indol-2yl amine-derived neuropeptide y receptor ligands useful in the treatment of obesity and other disorders

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington D.C. 20231 on

February 11, 2004 (Date of Deposit)

Ralph R, Palo

Name of Applicant, assignee or Registered

(Signature)

February 11, 2004 (Date of Signature)

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. 1.131

Dear Sir:

- 1. We, Scott L. Dax and James McNally declare that we are the inventors of the invention described and claimed in U.S. patent application Serial No. 09/522,969, filed on April 20, 2000, which application is based on provisional application Serial No. 60/132,660, filed May 5, 1999, and now abandoned.
- 2. We are presently, and were at and before completion of the invention, in the employ of Johnson & Johnson, which is the parent company of Ortho-McNeil Pharmaceutical Corporation, the assignee of record of the entire right, title and interest in the above-identified application.

- We are familiar with the Office Action dated June 6, 2003 in which the above referenced application was rejected over a publication by McNally et al. which appeared in Bioorganic and Medicinal Chemistry Letters 10 (2000) 213-216.
- We declare that the invention described and claimed in the above identified application was conceived by us in this country prior to February 7, 2000 and that such conception was coupled with due diligence by us in this country from just prior to February 7, 2000 to a reduction to practice of the invention.
- 5. Exhibits A-E attached hereto consist of true copies from which the dates have been removed of documents from the Ortho-McNeil Pharmaceutical Corporation research laboratories which record the generic conception of the invention.
- 6. Exhibits F-AA attached hereto consist of true copies from which the dates have been removed of documents from the Crystalytics Company which illustrate the reduction to practice of the invention.
- Exhibits AB-AQ attached hereto consist of true copies from which the dates have been removed of documents from the Ortho-McNeil Pharmaceutical Corporation research laboratories which illustrate the operativeness of the invention. The assays and methods employed are known procedures which were published prior to February 7, 2000.
- All of the above procedures and tests were carried out by us or at our direction from a period prior to February 7, 2000 to a reduction to practice of the invention. The results of the testing indicated that the compounds were useful in the treatment of obesity and other disorders.
- We, Scott L. Dax and James McNally, further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

ate: $\frac{2/10/2004}{2/10/2004}$

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Jim McNally
Exhibit F

CRYSTAL STRUCTURE ANALYSIS REPORT and TABLES for

 $[C_{27}H_{36}N_3O_3S][Cl]$

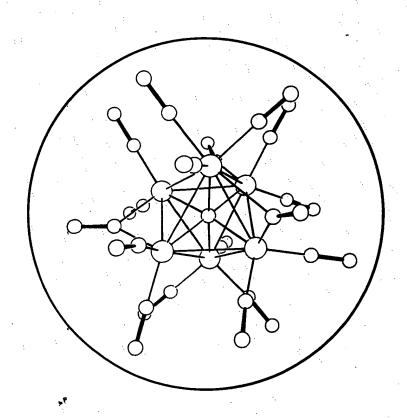
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For: The R. W. Johnson Research Institute

Sample #14794-65-1

Jim McNally

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CRYSTALYTICS COMPANY

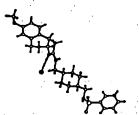
CRYSTAL STRUCTURE CONSULTING

BRIEF EXPERIMENTAL DESCRIPTION TO BE INCLUDED IN TEXT OR AS A FOOTNOTE AT TIME OF PUBLICATION

Single crystals of $[C_{27}H_{36}N_3O_3S][Cl]$ are, at 20 ± 1 °C, orthorhombic, space group Pna2₁ - C_{2v}^{9} (No. 33) with $\underline{a} = 42.245(1)$ Å, $\underline{b} = 5.3262(2)$ Å, $\underline{c} = 11.9748(4)$ Å, V = 2694.4(2) Å³, and $Z = 4 \{d_{calcd} = 1.277 \text{ gcm}^{-3}; \mu_a(CuK\tilde{\alpha}) = 2.24 \text{ mm}^{-1}\}$. A full hemisphere of diffracted intensities (omega or phi scans with width of 0.25°) was measured using graphitemonochromated CuKα radiation on a Siemens X-1000 HI-STAR Multiwire area detector. X-rays were provided by a Siemens M18XHF rotating anode operated at 40kV and 70mA. The sample was a nonmerohedrally twinned specimen containing two domains; the major domain had approximately 17 times the volume of the minor domain and only 4% of the reflections for the two domains were partially overlapped. There were no totally overlapping reflections and partially overlapping reflections were not used for structure refinement. The diffraction data from each domain was used to solve the structure independently and both gave the same species. The results reported herein are for the major domain but nearly identical structural parameters resulted from refinement using the nonoverlapping data for both domains simultaneously. Structure refinement using just the data from the minor domain gave structural parameters with reduced precision.

Lattice constants for the major domain were determined with the Siemens SAINT software package using peak centers for 2292 reflections. A total of 4902 integrated reflection intensities having $2\Theta(\text{CuK}\alpha) < 104.3^{\circ}$ were produced using the Siemens program SAINT. A total of 2788 of these were independent and gave $R_{\text{int}} = 0.057$. The Siemens SHELXTL-PC software package was used to solve the structure using "Direct Methods" techniques. All stages of weighted full-matrix least-squares refinement were conducted using F_0^2 data and the SHELXTL-PC Version 5 software package and converged to give R_1 (unweighted, based on F) = 0.053 for 2223 independent absorption-corrected reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and $I > 2\sigma(I)$ and wR_2 (weighted, based on F^2) = 0.145 for 2673 independent absorption-corrected reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R values for all 2788 independent absorption-corrected reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and I > 0. Final R reflections having $2\Theta(\text{CuK}_{\alpha}) < 104.3^{\circ}$ and $2\Theta(\text{CuK}_{\alpha}) <$

atoms and isotropic thermal parameters for all hydrogen atoms. Hydrogen atoms H_{1N} , H_{2N} and H_{3N} were located from a difference Fourier map and refined as independent isotropic atoms. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp²- or sp³-hybridization of the carbon atoms and C-H bond lengths of 0.93-0.98 Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{1N} , H_{2N} and H_{3N} refined to final U_{iso} values of 0.10(3), 0.11(3) and 0.01(2)Å², respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (nonmethyl) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded. The methyl group (C_{27} and its hydrogens) was refined as a rigid rotor (using idealized sp³-hybridized geometry and a C-H bond length of 0.96 Å) with three rotational parameters in each least-squares cycle. The refined values of these rotational parameters gave O-C-H angles which ranged from 105° to 117°.



Crystalytics Company Crystal Structure Analysis Report

Compound Formula: [C₂₇H₃₆N₃O₃S][Cl]

Reference Code: CFS4-0799

R. W. J. sample #14794-65-1 (Jim McNally)

Description of Single-Crystal Sample and Mounting Used for Data Collection:

1) Color: Colorless

2) Shape: Flat plate

3) Dimensions: 0.025 mm, x 0.125 mm, x 0.175 mm.

4) Indices of Faces:

5) Crystal Mount: Crystal was glued with epoxy to the end of a thin glass fiber.

6) Crystal Orientation: Crystal was oriented with its longest edge nearly parallel to the phi axis of the diffractometer.

7) Comments: Sample was recrystallized from methanol/ethylacetate solution.

Space Group and Cell Data:

1) Crystal System: Orthorhombic Space Group and Number¹: Pna2₁ - C_{2v} (No. 33)

2) Number of Computer-Centered Reflections Used in the Least-Squares Refinement of the Cell Dimensions: 2292 measured at: 20±2 °C

3) Lattice Constants with esd's:

a = 42.245(1) Å $\alpha = 90.000^{\circ}$ $V = 2694.4(2) \text{ Å}^3$ b = 5.3262(2) Å $\beta = 90.000^{\circ}$ Z = 4 formula unitsc = 11.9748(4) Å $\gamma = 90.000^{\circ}$ $\lambda = 1.54178 \text{ Å}$

4) Molecular Weight: 518.10 amu/formula unit Calculated Density: 1.277 g cm⁻³

5) Linear Absorption Coefficient^{2a}: 2.24 mm^{-1} F(000) = 1104.

Comments: The sample was a nonmerohedrally twinned specimen containing two domains; the major domain had approximately 17 times the volume of the minor domain and only 4% of the reflections for the two domains were partially overlapped. There were no totally overlapping reflections and partially overlapping reflections were not used for structure refinement. The diffraction data from each domain was used to solve the structure independently and both gave the same species. The results reported herein are for the major domain but nearly identical structural parameters resulted from refinement using the nonoverlapping data for both domains simultaneously. Structure refinement using just the data from the minor domain gave structural parameters with reduced precision.

Reference Code: CFS4-0799

Description of Data Collection³:

1) Instrument: Bruker X-1000 HI-STAR Single Crystal Multiwire Diffraction System

2) X-ray Source: Bruker M18XHF Rotating Anode with 0.3 x 3.0 mm. filament

3) Radiation: CuKā Power: 40 kV 70 mA

4) X Monochromator: X Graphite Other (Specify:)
Filter: Nickel Niobium Other (Specify:)

5) Incident Beam Collimator Diameter: 0.5 mm Temperature: 20±2 °C

6) Scan Axis: X Omega or X Phi

7) Scan Width: 0.25° 20 Range of Data: 8.38° - 104.28°

8) Sample to Detector Distance: 8.56 cm

9) Portion of Ewald Sphere Collected: Hemisphere

10) Number of frames collected: 3228 Seconds/frame: 60

11) Total Number of Reflections Collected: 4902

12) Number of Independent Reflections Collected: 2788

13) Data Collected: $-28 \le h \le 43$; $-5 \le k \le 5$; $-11 \le l \le 12$ $R_{int}^{4} = 0.057$

Data Reduction³:

1) Lorentz and Polarization Corrections? Yes

2) Absorption Correction: Yes Range of transmission factors: 0.524 - 0.666
XX Empirical Correction using Measurements for Equivalent Reflections
(508 Reflections used)

___ Face-Indexed Gaussian Grid Correction

3) Comments:

Structure Solution⁵:

Method(s) Used in Structure Solution
 — Heavy-atom Patterson Techniques

XX Direct Methods

- a) XX SHELXTL/PC
- b) __Other

__Other Techniques

2) Hydrogen Atom Positions Located? Yes

After Refinement Cycle # 2 by XX Difference Fourier

XX Calculated

3) Comments:

Reference Code: CFS4-0799

Structure Refinement⁵: (see next page for summary of refinement cycles)

1) Final Scale Factor: 0.327(1)

2) Extinction Parameter⁶ Refined? Yes Final Value: 0.0002(2)

Form: $k[1+0.001(x)(F_c^2)(\lambda^3)/\sin(2\theta)]^{-1/4}$

3) Anomalous Dispersion Corrections^{2b} for Which Atoms: Cl, S, O, N, C

4) Variable Occupancies for Which Atoms? None

Atom

Final Occupancy

Atomic Form Factor^{2c} Used

- So Refinement Constraints/Restraints: Hydrogen atoms H_{1N}, H_{2N} and H_{3N} were located from a difference Fourier map and refined as independent isotropic atoms. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp²- or sp³-hybridization of the carbon atoms and C-H bond lengths of 0.93-0.98 Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{1N}, H_{2N} and H_{3N} refined to final U_{iso} values of 0.10(3), 0.11(3) and 0.01(2)Å², respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (nonmethyl) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded. The methyl group (C₂₇ and its hydrogens) was refined as a rigid rotor (using idealized sp³-hybridized geometry and a C-H bond length of 0.96 Å) with three rotational parameters in each least-squares cycle. The refined values of these rotational parameters gave O-C-H angles which ranged from 105° to 117°.
- 6) Shift/Error Analysis for Final Least-Squares Cycle⁷:
 Maximum Shift for all Parameters: <u>0.000</u> σ_p Mean Shift for all Parameters: <u>0.000</u> σ_p
- 7) Peaks found in Final Difference Fourier Map: There were no peaks present in the final difference Fourier map above the background level (0.35 e⁻/Å³). The minimum and mean electron density in the final difference Fourier were -0.19 and 0.00 e⁻/Å³, respectively. The rms deviation from the mean electron density was 0.04 e⁻/Å³.

CRYSTAL STRUCTURE ANALYSIS REPORT

Summary of Full Matrix Least-Squares Refinement⁸ Cycles

 	Extincti n	Т	T T		
	Correcti n			×	
d on F ²) ¹⁰	'Goodness- of-fit' (Goof) ¹³	1.755	1.400	1.045	
\underline{R}_2 (weighted,based on F^2) 10	Total # Independent Reflections	2673	2673	2673	·
	R ₂ 12	0.249	0.193	0.145	
ased on F)	F _o / σ(F _o). Cutoff	4.0	4.0	4.0	
R ₁ (unweighted,based on F)	# Observed Reflections	2223	2223	2223	
$\mathbf{R_1}$ (u	$\underline{\mathbf{R}_{\mathbf{l}}}^{11}$	0.093	0.074	0.053	
. :	# Refined Parameters	141	316	332	
s	Thermal Parameters	X		Х	
Atom	Positions Refined	×	:	×	
Isotropic Atom	Number and Type	27 C, 3 N 3 O, 1 S 1 Cl	:	. 36Н	
Anisotropic ⁹	Atoms Number and Type		27 C, 3 N 3 O, 1 S 1 Cl	27 C, 3 N 3 O, 1 S 1 Cl	
γ /ι	Maximum	0.51	0.51	0.51	
sin θ/ λ	Minimum	0.00	0.00	0.00	
Су	cle Number	-	2	3	

* See Item 5 on page 3 regarding the treatment of the hydrogen atoms.

Final Statistics from Cycle #3 for All of the Reflection Data: $R_1 = 0.074$; w $R_2 = 0.166$; GOOF = 1.169 for 2788 reflections

The correctness of the assigned absolute configuration was checked using the "Flack" absolute structure parameter 14 which refined to a final value of 0.18(3).

Reference Code: CFS4-0799

References and Notes

- 1. "International Tables for X-Ray Crystallography", Vol. A, Kluwer Academic Publishers, Dordrecht, 1995.
- 2. "International Tables for X-Ray Crystallography", Vol. C, Kluwer Academic Publishers, Dordrecht, 1992; a) Tables 4.2.4.2 pp. 193-199; b) Tables 4.2.6.8 pp 219-222; c) Tables 6.1.1.4 pp 500-502.
- 3. Data acquisition and reduction was accomplished using standard versions of Siemens/Bruker software for the diffraction system.
- 4. $R_{int} = \Sigma |F_0^2 F_0^2(mean)| / \Sigma [F_0]^2$
- 5. All structure determination and refinement calculations were performed on an IBM compatible 486 or 586 personal computer using the Siemens/Bruker SHELXTL Version 5.0 PC interactive software package.
- 6. A. C. Larson in "Crystallographic Computing", 1970, Ed. F. R. Ahmed, Munksgaard, Copenhagen, pp 291-294.
- 7. $\sigma_{\rm p}$ is the estimated standard deviation of the parameter in question.
- 8. Refinement on F^2 for all reflections except for 115 with negative F^2 . Weighted R-factors wR_2 and all goodnesses of fit S are based on F^2 , conventional R-factors R_1 are based on F, with F set to zero for negative F^2 . The observed criterion of $F^2 > 2 \operatorname{sigma}(F^2)$ is used only for calculating "R-factor obs" etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on all data will be even larger.
- 9. The anisotropic thermal parameter is of the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)].$
- 10. The weighting scheme used is defined as: $w = 1 / [\sigma^2(F_0^2) + (a^*P)^2 + b^*P + d + e^*\sin(\theta)]$ where $P = [F_0^2 + 2F_c^2]/3$. In this case, $a = \underline{0.0938}$, $b = \underline{0}$, $d = \underline{0}$ and $e = \underline{0}$.
- 11. $\underline{R}_1 = \Sigma ||F_0| |F_c||/\Sigma |F_0|$
- 12. $w\underline{R}_2 = \left[\Sigma[w(F_o^2 F_c^2)^2]/\Sigma[w(F_o^2)^2]\right]^{\frac{1}{2}}$
- 13. GooF = S = $\left[\sum \left[w(F_0^2 F_c^2)^2\right]/(n-p)\right]^{1/2}$ where n is the total number of reflections and p is the number of parameters refined.
- 14. The value of the "Flack absolute structure parameter", x, should be 0.00 for the correct enantiomorphic description and 1.00 for the inverted description: a) H. D. Flack, Acta Cryst., 1983, A39, 876-881; b) G. Bernardinelli and H. D. Flack, Acta Cryst., 1985, A41, 500-511.

Table 1. Atomic Coordinates for Nonhydrogen Atoms in Crystalline [C₂₇H₃₆N₃O₃S][Cl] ^a

·	·	<u> </u>	<u>.</u>		
Atom	Fra	ctional Coordinate		Equivalent Isotropic Thermal Parameter,	
Type ^b	10 ⁴ x	10 ⁴ y	10 ⁴ z	U, $Å^2 \times 10^3$ c	٠
	· ·	Cation			
S	1890(1)	-1817(3)	3378(2)	62(1)	
O ₁	1739(1)	-4228(8)	3392(5)	84(1)	
O_2	2083(1)	-1058(9)	4292(4)	79(1)	
O ₃	-1850(1)	7660(12)	3106(5)	105(2)	÷
N_1	-362(1)	2030(13)	2990(6)	62(2)	:
N_2	-53(1)	5255(12)	2246(6)	66(2)	
N ₃	1619(2)	209(16)	3278(7)	67(2)	:
C_1	-297(2)	3645(14)	2220(6)	59(2)	
C_2	-525(2)	3482(13)	1278(5)	65(2)	
C_3	-793(2)	1762(14)	1736(6)	67(2)	
C ₄	-1083(2)	3353(14)	2115(6)	64(2)	
C ₅	-1248(2)	4655(17)	1347(7)	86(2)	:
C_6	-1510(2)	6149(16)	1651(8)	87(3)	
$\overset{\circ}{C_7}$	-1598(2)	6274(16)	2739(8)	77(2)	•
C ₈	-1441(2)	4911(14)	3526(7)	73(2)	
C ₉	-1175(1)	3462(11)	3245(6)	55(2)	
C ₁₀	-996(2)	2072(16)	4119(6)	77(2)	5 5
C ₁₁	-829(2)	-256(15)	3679(6)	75(2)	·.
C ₁₂	-629(2)	351(13)	2681(6)	61(2)	
C ₁₃	186(2)	5209(14)	3088(6)	66(2)	
C ₁₄	443(1)	3309(12)	2830(5)	53(2)	
C ₁₅	630(2)	4008(13)	1781(6)	62(2)	
C ₁₆	906(1)	2211(14)	1576(5)	60(2)	
C ₁₇	1121(1)	2009(12)	2563(5)	52(2)	
C ₁₈	931(1)	1224(13)	3608(5)	59(2)	-
C ₁₉	663(2)	3025(14)	3812(5)	64(2)	
19	. ` ′	• • • •			

(continued) Table 1.

Atom	Fr	actional Coordina	Equivalent Isotropic	
Type ^b	10 ⁴ x	10 ⁴ y	10 ⁴ z	Thermal Parameter, U, Å ² x 10 ³ c
C ₂₀	1388(1)	186(14)	2353(6)	63(2)
C ₂₁	2110(2)	-1655(13)	2128(6)	60(2)
C ₂₂	2064(2)	-3338(17)	1289(8)	94(3)
C ₂₃	2237(2)	-3108(20)	305(9)	111(3)
C ₂₄	2456(2)	-1242(21)	189(9)	105(3)
C ₂₅	2502(2)	386(19)	1015(9)	102(3)
C ₂₆	2332(2)	215(17)	1990(8)	90(3)
C ₂₇	-1972(2)	9523(21)	2397(10)	122(4)
21		Anion	. :	
Cl	-64(1)	744(4)	5362(2)	75(1)

The numbers in parentheses are the estimated standard deviations in the last significant digit. Atoms are labeled in agreement with Figure 1. This is one-third of the trace of the orthogonalized U_{ij} tensor.

Table 2. Anisotropic Thermal Parameters for Nonhydrogen Atoms in Crystalline $[C_{27}H_{36}N_3O_3S][Cl]^{a,b}$

	Atom	•	Anisotropic Thermal Parameters (Å ² x 10 ³)				TT
	Type ^c	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
•							
			.*	Cation			a (4)
	S	59(1)	55(1)	74(1)	6(1)	-14(1)	-3(1)
	O_1	82(3)	48(3)	121(4)	21(3)	-10(3)	-13(2)
	O_2	81(3)	79(3)	77(3)	6(3)	-22(3)	-15(3)
	O_3	79(3)	116(5)	120(6)	20(4)	21(4)	29(3)
•	N_1	58(4)	73(4)	56(4)	4(4)	0(3)	6(3)
	N ₂	56(4)	56(4)	86(5)	4(4)	8(3)	4(3)
	N ₃	68(4)	61(5)	73(5)	-11(5)	-12(3)	-17(4)
	C_1	64(5)	69(5)	44(4)	13(4)	1(4)	22(4)
	C_2	62(4)	74(5)	60(4)	4(4)	8(4)	16(4)
•	C ₃	51(4)	71(5)	79(5)	-15(4)	15(4)	12(4)
	C_4	61(4)	66(5)	66(5)	8(4)	-11(4)	-10(4)
	C ₅	78(5)	108(7)	74(5)	3(5)	-15(5)	18(5)
:	C_6	68(5)	96(7)	96(7)	27(5)	-10(5)	19(5)
	C_7	41(4)	95(6)	95(6)	19(5)	0(4)	10(4)
	C ₈	60(4)	76(5)	84(5)	4(5)	7(4)	-8(4)
	C ₉	47(3)	57(4)	60(5)	9(4)	1(3)	1(3)
	C ₁₀	69(5)	91(6)	72(5)	10(5)	7(4)	-12(5)
	C ₁₁	67(4)	80(5)	77(5)	10(5)	-15(4)	-6(4)
	C ₁₂	56(4)	56(4)	72(5)	-5(4)	-3(4)	4(4)
	C ₁₂	55(4)	68(4)	75(5)	-6(4)	3(4)	2(3)
•	C ₁₄	47(3)	52(4)	60(4)	2(3)	7(3)	7(3)
	C ₁₅	57(4)	60(4)	69(5)	2(4)	4(4)	3(4)
		54(4)	67(5)	58(4)	-1(4)	9(3)	7(3)
	C ₁₆	50(4)	54(4)	52(4)	0(3)	4(3)	0(3)
	C ₁₇	52(4)	72(5)	53(4)	0(4)	0(3)	3(3)
	C ₁₈		80(5)	47(4)	3(4)	6(3)	-13(4)
	C ₁₉	66(4)	69(5)	68(5)	-9(4)	-2(4)	7(4)
	C ₂₀	50(4)	03(3)	30(3)		` '	

(continued) Table 2.

Atom		Anisotr	opic Thermal P	arameters (Å	$^{2} \times 10^{3}$)	
Type ^c	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
C ₂₁	57(4)	47(4)	75(5)	3(4)	-1(4)	0(4)
C ₂₂	84(6)	87(6)	109(7)	-34(6)	11(5)	-8(5)
C ₂₃	105(7)	110(8)	118(8)	-33(7)	40(7)	0(6)
C ₂₄	104(7)	104(7)	108(8)	-21(7)	27(6)	7(6)
C ₂₅	96(6)	100(7)	109(8)	-7(7)	33(6)	-23(6)
C ₂₆	92(6)	80(6)	99(7)	-12(5)	28(5)	-21(5)
C ₂₇	97(7)	103(8)	164(11)	14(8)	-ó(7)	33(6)
21			Anion			
Cl	85(1)	75(1)	65(1)	11(1)	-10(1)	-1(1)

The numbers in parentheses are the estimated standard deviations in the last significant digit. The form of the anisotropic thermal parameter is: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$. Atoms are labeled in agreement with Figure 1.

Table 3. Atomic Coordinates for Hydrogen Atoms in Crystalline $[C_{27}H_{36}N_3O_3S][Cl]^a$

Atom Type ^b	10 ⁴ x	Fractional Coordinates 10 ⁴ y	10 ⁴ z
H _{1N} ^c	-288(20)	1722(159)	3622(80)
H _{2N} ^c	1(18)	6723(161)	1614(80)
H _{3N} ^c	1657(12)	1194(90)	3415(53)
H_{2b}	-427	2747	623
H _{2c}	-607	5129	1086
H ₃	-858	573	1155
H_5	-1187	4570	601
H ₆	-1622	7037	1110
H ₈	-1511	4938	4262
H _{10a}	-1141	1579	4707
H _{10b}	-839	3188	4445
H _{11a}	-696	-957	4263
H _{11b}	-985	-1509	3476
H ₁₂	-542	-1217	2382
H _{13a}	89	4797	3800
H _{13b}	279	6865	3152
H ₁₄	342	1683	2697
H _{15a}	490	3978	1140
H _{15b}	711	5703	1859
H _{16a}	824	561	1397
H _{16b}	1027	2795	938
H ₁₇	1213	3668	2704
H _{18a}	847	-455	3504
H _{18b}	1070	1200	4253
H _{19a}	750	4658	3997
H _{19b}	542	2449	4450
H _{20a}	1302	-1493	2271
H _{20b}	1494	628	1662

Table 3. (continued)

Atom Type ^b	10 ⁴ x	Fractional Coordinates 10 ⁴ y	10 ⁴ z
H ₂₂	1918	-4630	1371
H ₂₃	2204	-4234	-276
H ₂₄	2572	-1109	-467
H ₂₅	2652	1655	931
H ₂₆	2367	1368	2561
H _{27a}	-2103	10560	2860
H _{27b}	-1822	10551	2008
H _{27c}	-2101	8649	1868

Hydrogen atoms covalently bonded to nitrogens (H_{1N}, H_{2N} and H_{3N}) were located from a difference Fourier map and refined as independent isotropic atoms. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp²- or sp³-hybridization of the carbon atoms and C-H bond lengths of 0.93 Å to 0.98 Å) "riding" on their respective carbon atoms. The methyl group (C₂₇ and its hydrogens) was refined as a rigid rotor (using idealized sp³-hybridized geometry and a C-H bond length of 0.96 Å) with three rotational parameters in each least-squares cycle. The refined values of these rotational parameters gave O-C-H angles which ranged from 105° to 117°. The isotropic thermal parameters for H_{1N}, H_{2N} and H_{3N} refined to final U_{iso} values of 0.10(3), 0.11(3) and 0.01(2) Å², respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (nonmethyl) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded.

Hydrogen atoms which are covalently bonded to carbon are labeled with the same numerical subscript(s) as their carbon atoms with an additional literal subscript (a, b or c) where necessary to distinguish between hydrogens bonded to the same carbon atom. The amine hydrogen atoms are labeled H_{1N} and H_{2N} and the sulfonamide hydrogen atom is labeled H_{3N}.

The numbers in parentheses are the estimated standard deviations in the last significant digit.

Table 4. Bond Lengths in Crystalline [C₂₇H₃₆N₃O₃S][Cl] ^a

Type ^b	Length, Å	Type ^b	Length, Å	. *
S-O ₁	1.434(4)	S-O ₂	1.423(5)	
S-N ₃	1.579(8)	S-C ₂₁	1.763(7)	÷
O ₃ -C ₇	1.368(9)	O ₃ -C ₂₇	1.404(11)	
N ₁ -C ₁	1.291(9)	C_1 - C_2	1.487(9)	٥
N ₂ -C ₁	1.340(9)	C ₉ -C ₁₀	1.489(10)	· :
N ₁ -C ₁₂	1.487(9)	N ₁ -H _{1N}	0.84(9)	
$N_1 C_{12}$ $N_2 C_{13}$	1.425(9)	N ₂ -H _{2N}	1.11(9)	
N_3-C_{20}	1.477(10)	N_3-H_{3N}	0.57(5)	
			:	
C_2 - C_3	1.555(9)	C_4 - C_5	1.346(10)	:
$C_3 - C_{12}$	1.525(10)	C ₄ -C ₉	1.408(10)	
C_3-C_4	1.557(10)	C ₅ -C ₆	1.413(11)	•
C ₁₀ -C ₁₁	1.520(10)	C ₆ -C ₇	1.356(12)	
C ₁₁ -C ₁₂	1.498(10)	C ₇ -C ₈	1.362(10)	÷,
C ₁₃ -C ₁₄	1.517(9)	C ₈ -C ₉	1.406(9)	•
$C_{14}-C_{19}$	1.505(9)	C_{21} - C_{22}	1.361(10)	· .·
C ₁₄ -C ₁₅	1.531(9)	C_{21} - C_{26}	1.377(10)	
C ₁₅ -C ₁₆	1.529(9)	C ₂₂ -C ₂₃	1.392(13)	·
$C_{16}-C_{17}$	1.493(9)	C ₂₃ -C ₂₄	1.364(13)	
$C_{17}C_{20}$	1.509(9)	C_{24} - C_{25}	1.329(12)	
C ₁₇ -C ₁₈	1.544(9)	C ₂₅ -C ₂₆	1.375(12)	
C ₁₈ -C ₁₉	1.504(9)			٠.

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.

b Atoms are labeled in agreement with Figure 1.

Table 5. Bond Angles in Crystalline [C₂₇H₃₆N₃O₃S][Cl] ^a

Type ^b	Angle, (deg)	Type ^b	Angle, (deg)
O ₂ SO ₁	120.0(3)	O ₂ SC ₂₁	109.8(3)
O_2SN_3	106.2(4)	O_1SC_{21}	106.8(4)
O_1SN_3	106.8(3)	N_3SC_{21}	106.5(4)
1 3			
C ₇ O ₃ C ₂₇	118.2(7)	$C_1N_2C_{13}$	123.2(7)
, 3 21		$C_{20}N_3S$	122.1(6)
$C_1N_1C_{12}$	112.7(7)	:	· •
	• •		
$C_1N_1H_{1N}$	134(6)	$C_{13}N_2H_{2N}$	111(4)
$C_{12}N_1H_{1N}$	113(6)	$C_{20}N_3H_{3N}$	114(6)
$C_1N_2H_{2N}$	126(4)	SN_3H_{3N}	114(6)
·			:
$N_1C_1N_2$	125.0(7)	$N_1C_1C_2$	111.4(7)
$N_2C_1C_2$	123.6(6)		
$C_1C_2C_3$	103.8(6)	$C_{19}C_{14}C_{15}$	110.3(5)
$C_{12}C_3C_2$	102.9(5)	$C_{13}C_{14}C_{15}$	112.0(5)
$C_{12}C_3C_4$	114.1(6)	$C_{16}C_{15}C_{14}$	112.0(5)
$C_2C_3C_4$	110.7(6)	$C_{17}C_{16}C_{15}$	112.4(5)
$C_9C_{10}C_{11}$	113.4(6)	$C_{16}C_{17}C_{20}$	111.6(5)
$C_{12}C_{11}C_{10}$	111.2(6)	$C_{16}C_{17}C_{18}$	110.3(5)
$N_1C_{12}C_{11}$	111.1(6)	$C_{20}C_{17}C_{18}$	110.4(5)
$N_1C_{12}C_3$	103.4(6)	$C_{19}C_{18}C_{17}$	110.5(5)
$C_{11}C_{12}C_3$	116.3(5)	$C_{18}C_{19}C_{14}$	113.7(5)
$N_2C_{13}C_{14}$	112.0(6)	$N_3C_{20}C_{17}$	111.4(6)
$C_{19}C_{14}C_{13}$	110.5(5)		
$C_5C_4C_9$	119.5(7)	$C_8C_9C_{10}$	120.8(7)

(continued) Table 5.

Type ^b	Angle, (deg)	Type ^b	Angle, (deg)
$C_5C_4C_3$	119.2(7)	$C_4C_9C_{10}$	121.0(6)
$C_9C_4C_3$	121.3(6)	$C_{22}C_{21}C_{26}$	119.0(7)
$C_4C_5C_6$	121.4(8)	$C_{22}C_{21}S$	121.3(6)
$C_7C_6C_5$	119.3(7)	$C_{26}C_{21}S$	119.7(6)
$C_6C_7C_8$	120.4(7)	$C_{21}C_{22}C_{23}$	119.5(8)
$C_6C_7O_3$	123.2(8)	$C_{24}C_{23}C_{22}$	120.4(10)
$C_8C_7O_3$	116.3(8)	$C_{25}C_{24}C_{23}$	120.0(9)
$C_7C_8C_9$	121.1(8)	$C_{24}C_{25}C_{26}$	120.7(9)
$C_8C_9C_4$	118.3(6)	$C_{25}C_{26}C_{21}$	120.4(9)

The numbers in parentheses are the estimated standard deviations in the last significant digit.

Atoms are labeled in agreement with Figure 1.

Hydrogen-Bonding Interactions in Crystalline [C₂₇H₃₆N₃O₃S][Cl] Table 6.

Donor Atom (D) ^a	Acceptor Atom (A)	Distance Å D···A	Distance Å H A	Angle deg. D-H···A	Angle deg. H-D A	Angle deg. H···A-X ^b	Asymmetric Unit of A ^c
N ₁ -H _{1N}	Cl	3.183	2.35	178	2	124(H ₂₁	_N) x, y, z
N_2 - H_{2N}	Cl	3.142	2.03	174	4	124(H ₁)	N) -x, 1-y, $-0.5 + z$
N_3 - H_{3N}	O_1	3.009	2.46	161	16	162(S)	x, 1+y, z

The hydrogen atom involved in the interaction is also indicated.

The symbol X is used to denote the atoms which are covalently or hydrogen bonded to the b acceptor atoms.

All donor atoms belong to the asymmetric unit for which fractional atomic coordinates are given in Tables 1 and 3.

FIGURE CAPTIONS

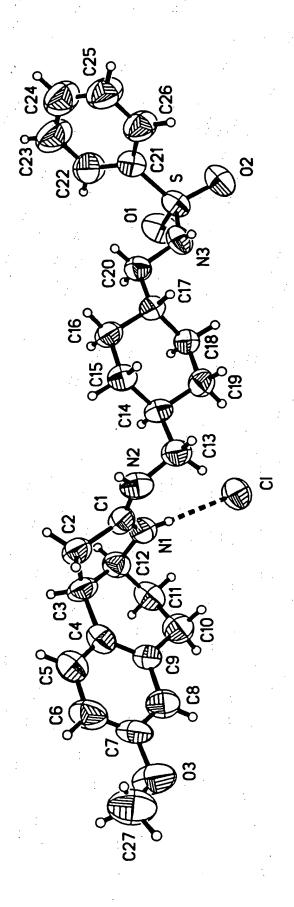
- Figure 1a. shows a perspective drawing of the solid-state structure for the HCl salt of [C₂₇H₃₅N₃O₃S]. Nonhydrogen atoms are represented by 50% probability thermal vibration ellipsoids and hydrogen atoms are represented by arbitrarily-small spheres which are in no way representative of their true thermal motion. The hydrogen-bonding interaction between the Cl⁻ anion and the hydrogen on amine nitrogen N₁ is shown with a dashed line.
- Figure 1b. shows a perspective drawing of the solid-state structure for the HCl salt of [C₂₇H₃₅N₃O₃S]. The view is the same as in Figure 1a but the chlorine atom is now represented as a large dotted sphere, oxygen and nitrogen atoms are now represented by medium-sized shaded spheres, and sulfur, carbon and hydrogen atoms are represented by large, medium and small open spheres, respectively. The hydrogen-bonding interaction between the Cl anion and the hydrogen on amine nitrogen N₁ is shown with a dashed line.
- Figure 1c. shows a perspective drawing of the structure for the $[C_{27}H_{36}N_3O_3S]^+$ cation, as observed in the solid-state structure of its Cl salt. Nonhydrogen atoms are represented by 50% probability thermal vibration ellipsoids and hydrogen atoms are represented by arbitrarily-small spheres which are in no way representative of their true thermal motion.
- Figure 1d. shows a perspective drawing of the structure for the $[C_{27}H_{36}N_3O_3S]^+$ cation, as observed in the solid-state structure of its Cl^- salt. The view is the same as in Figure 1c but the oxygen and nitrogen atoms are now represented by medium-sized shaded spheres, and sulfur, carbon and hydrogen atoms are represented by large, medium and small open spheres, respectively.
- Figure 1e. shows a space-filling drawing of the structure for the [C₂₇H₃₆N₃O₃S]⁺ cation, as observed in the solid-state structure of its Cl⁻ salt. The view is as in Figure 1c.

Figure 1f. shows

shows a perspective drawing of the hydrogen-bonding interaction involving the Clanion and the amine $(N_1 \text{ and } N_2)$ nitrogens of two symmetry-related $[C_{27}H_{36}N_3O_3S]^+$ cations in the crystal. Atoms of the cation related by symmetry operation -x, 1-y, -0.5+z to the one shown in Figure 1d are labeled with a prime ('). Atoms are represented as in Figure 1b and the hydrogen bonds between the Clanion and the $[C_{27}H_{36}N_3O_3S]^+$ cations are shown with dashed lines.

Figure 1g.

shows a perspective drawing of the hydrogen-bonding interaction involving sufonamide proton H_{3N} and the sulfonamide oxygen atom of a symmetry-related $[C_{27}H_{36}N_3O_3S]^+$ cation in crystals of $[C_{27}H_{36}N_3O_3S][Cl]$. Atoms of the $[C_{27}H_{36}N_3O_3S]^+$ cation related by symmetry operation x, 1+y, z to the one shown in Figure 1d are labeled with double primes ("). Atoms are represented as in Figure 1b and the hydrogen bond between $[C_{27}H_{36}N_3O_3S]^+$ cations is shown with a dashed line as are the hydrogen bonds between the cation and Cl^- anion within each of the two asymmetric units shown.



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